



TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

Haze measurement techniques

POC: Elena Bankowski

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Report Documentation Page

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1. Definition of haze

- Haze is defined as an aggravated form of fog in a polished surface caused by the scattering of light.
- In plastic industry, haze is a term used to describe the slightly cloudy appearance of film or sheet material resulting from forward scattering of light.
- Haze may arise from internal causes, such as dirt, poorly dispersed pigments, or other small particles within the specimen, or from external causes, such as surfaces with very fine roughness.
- The defects causing haze are larger than those causing fog, but singly are not large enough to be seen by unaided eye.
- It is desirable to have an objective measurement of haze, correlating with its visual perception, for quality control purposes.
- Our objective is to measure haze and understand what is causing it.
- This work is done in support of transparent armor specifications development.
- A method was devised using the integrating sphere to measure the forward scattering of light when the specimen was placed flush against the entrance port of the sphere.
- This method was adopted by the ASTM as the ASTM D 1003 standard and has been practiced ever since 1961.

Figure 1. Comparison of CIE illuminants A and C and the photopic response spectra

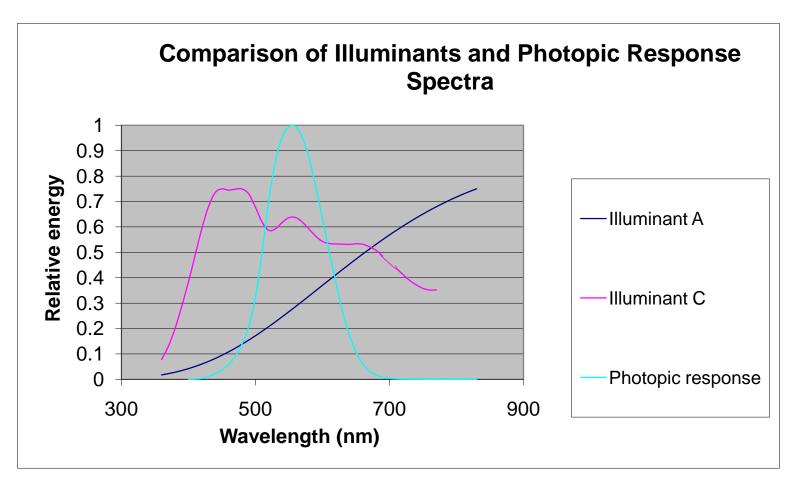


Figure 2. CIE illuminants A and C relative energy spectra multiplied by the CIE photopic response spectrum

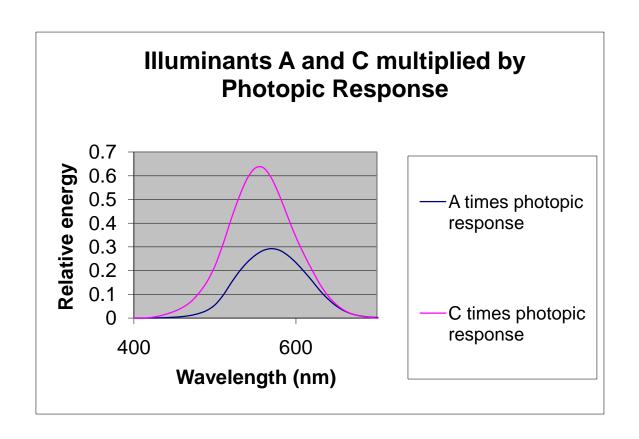
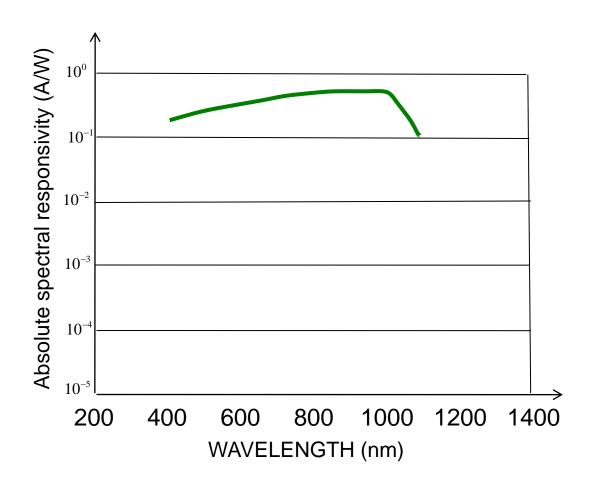


Figure 3. 818-SL silicon photodetector responsivity curve



III. Test procedure: Determine the following four readings:

| Reading Designation | Specimen in Position | Light Trap in Position | Reflectance Standard in Position | Quantity Represented |
|---------------------|----------------------|---------------------------|--|--|
| T1 | No | No | Yes | incident light |
| T2 | Yes | No | Yes | total light transmitted by specimen |
| Т3 | No | Yes | No | light scattered by instrument |
| T4 | Yes | Yes | No | light scattered by instrument and specimen |

IV. Calculation of Haze

Total Transmittance:

$$T_t = T_2 / T_1$$

Diffuse Transmittance:

$$T_d = [T_4 - T_3(T_2/T_1)]/T_1$$

Calculate percent haze as follows:

$$haze = T_d / T_t \times 100\%$$

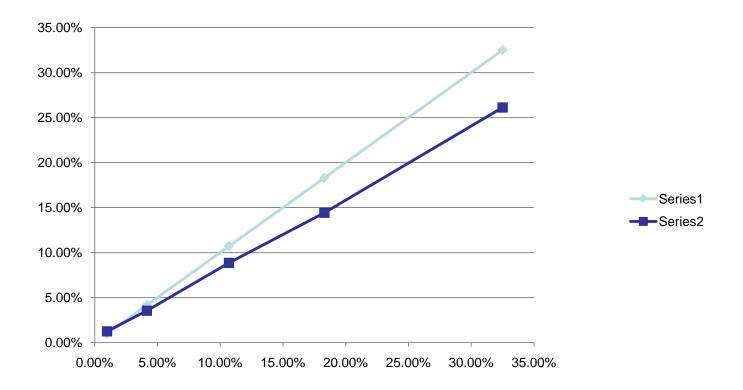
$$haze = (T_4 / T_2 - T_3 / T_1) \times 100\%$$

3. Haze measurement results

Haze standards measurements using 6" integrating sphere, CIE illuminant A, infrared filter and green filter # 102, reflectance standard, entrance beam diameter 10 mm and exit beam diameter 18 mm

| T1 | T2 | Т3 | T4 | Haze(%) | H(stand) | Error | Relative error | Abs(R. error) |
|-------|-------|-------|-------|---------|-------------------------------|--------|----------------|---------------|
| | | | | | | | | |
| 1.355 | 1.22 | 0.003 | 0.018 | 1.25% | 0.98% | -0.27% | -27.96% | 27.96% |
| 1.35 | 1.247 | 0.003 | 0.047 | 3.55% | 4.16% | 0.61% | 14.74% | 14.74% |
| 1.00 | 1.217 | 0.000 | 0.017 | 0.0070 | 1.1070 | 0.0170 | 1 117 170 | 11.7170 |
| 1.352 | 1.245 | 0.003 | 0.113 | 8.85% | 10.70% | 1.85% | 17.25% | 17.25% |
| | | | | | | | | |
| 1.355 | 1.229 | 0.003 | 0.18 | 14.42% | 18.30% | 3.88% | 21.18% | 21.18% |
| | | | | | | | | |
| 1.353 | 1.215 | 0.003 | 0.32 | 26.12% | 32.50% | 6.38% | 19.64% | 19.64% |
| | | | | | The average of values of rela | | | 18.20% |

3a. Haze measurement results



Series 1 – certified haze standards values % Series 2 – measured haze values %

4. Analysis of our haze measurement results.

- Analysis shows that the haze measurements are consistent only if the white standard has the same reflectance factor as the sphere wall (Fred W. Billmeyer, Y. Chent, 1985).
- This is the case in commercial hazemeters, but is unlikely to be true in the test with the integrating sphere.
- In the pivotable-sphere type instruments the reflectance is not changed in the measurements;
 the light beam is merely moved with respect to exit port.
- The reflectance factor can vary, among the standards in common use from 100% to 75%.
- I measured the reflectance factor of our standard; it was 99%.
- From visual observation, the inside surface of our integrating sphere appeared less reflective.
- I tried to reduce the reflectance of our standard in order to match the reflectance of our integrating sphere.
- I first covered it with a piece of white printer paper with the reflectance of 77%. This reduced the relative error of haze measurements to 5%.
- Next we made aluminum plugs for the exit port of the integrating sphere and painted two
 plugs with flat white primer paint. The reflectance of the first plug was 76.43%; the
 second was 81%.
- The haze measurements results improved, when we used the plug with reflectance factor of 76.43%. The average error of measurements was approximately 1%.

5. Haze standards measurements: new values of standards measured with a commercial instrument

| Certified old values | New values of Haze standards |
|----------------------|-------------------------------|
| of Haze standards | measured with Haze Guard Plus |
| 0.98% | 1.60% |
| 4.16% | 4.72% |
| 10.70% | 11% |
| 18.30% | 18.50% |
| 32.50% | 32.80% |

5. Brand new haze standards measured with a commercial tool

| Certified haze values | Values of new haze standards |
|-----------------------|---------------------------------------|
| of new haze standards | measured with a commercial instrument |
| | |
| 0.63% | 0.63% |
| 4.83% | 4.85% |
| | |
| 11.70% | 11.80% |
| 20.00% | 24.40/ |
| 20.90% | 21.1% |
| | |
| 29.0% | 29.3% |

Average Error of haze measurements was = 0.12%

6 a. Commercial device



6 b. Commercial device with abrasion holder



The measurement of haze is used to determine abrasion resistance of transparent materials. The Abrasion Holder facilitates positioning of the abraded area in the measurement beam.

7. Comparison of a commercial device and haze measurements using integrating sphere

- •Commercial device that was used demonstrated very high accuracy of haze measurements: +/- 0.01%.
- •Commercial device that was used was capable of measuring values of haze that are above 30%. If a haze value is above 30, the sample is so strongly scattering, that it is beyond hazy. These types of materials are usually referred to as diffusive. If we report values of haze above 30%, we should footnote that this is beyond the generally accepted range of what is considered to be "haze".
- •The distance between the clarity and haze ports is about 5", which is sufficient for most transparent armor samples we have seen thus far.
- •Abrasion holder for the commercial instrument creates a 7 mm beam.

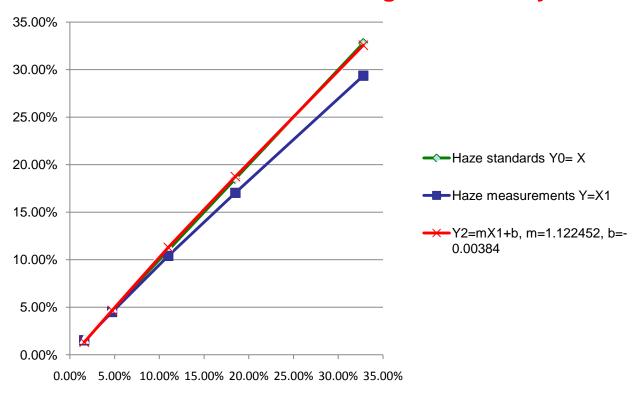
7. Comparison of commercial device and haze measurements using integrating sphere (continued)

- •With Abrasion Holder aperture in place, does the system still meet the ASTM requirements for beam size, divergence angle etc., -Yes to all; the system has to be re-calibrated with Abrasion Holder aperture in place.
- •We need to make sure that thick samples are normal to the beam to avoid measurement errors that can result from beam deviation.
- •Several fixtures are available to measure various samples. I am presently checking to determine if a fixture for thick and heavy samples is available to support the weight and not to deflect the bottom of the tool, which could deviate the optical axes of the sphere and the light source. We would need the fixture to make adjustments to ensure that the sample is inserted normal to the beam.
- •We saw that the haze, clarity and transparency values that were measured were strongly dependent upon the sample location. These values may be trusted for thin samples placed at the correct port during measurement, but can't be taken at face value without further interpretation for thick samples.
- •In the next phase of experiments, we'll be looking at the influence of beam size, sample placement, etc. on each of the 4 individual readings that make up the haze value.
- •Commercial haze measurements devices provide results that are industry accepted, but the user needs to recognize its limitations (e.g. thin samples placed right at the appropriate port, etc.).

8. Haze standards measurements using new values of standards obtained with commercial tool

| beam diameter 14 mm | | | | | | |
|---------------------|----------|-------|--------|--|--|--|
| Haze(measured) | H(stand) | Error | Error | | | |
| 1.53% | 1.60% | 0.07% | 4.42% | | | |
| 4.50% | 4.72% | 0.22% | 4.64% | | | |
| 10.41% | 11.00% | 0.59% | 5.34% | | | |
| 17.04% | 18.50% | 1.46% | 7.88% | | | |
| 29.36% | 32.80% | 3.44% | 10.48% | | | |

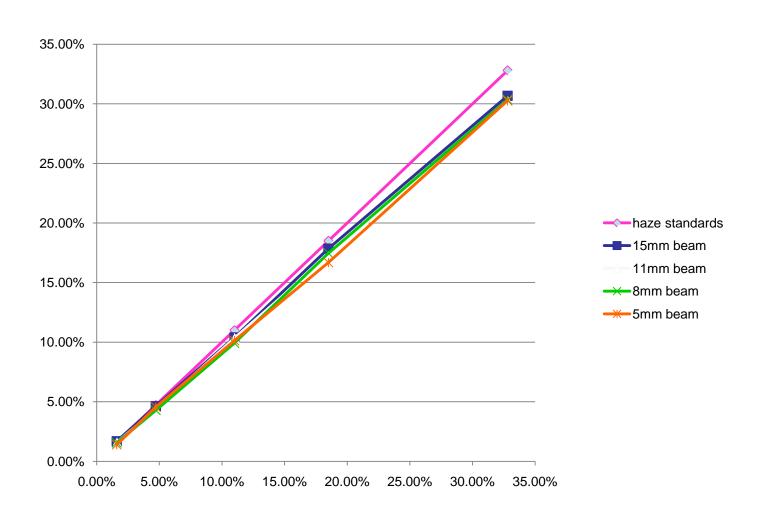
9. Comparison of haze measurements with new values of standards obtained with commercial tool and regression analysis



10. Comparison of calculated haze values with those obtained with commercial tool

| beam | | Relative | |
|------------------|----------|----------|--------|
| Haze(calculated) | H(stand) | Error | Error |
| 1.33% | 1.60% | 0.27% | 16.71% |
| 4.67% | 4.72% | 0.05% | 1.09% |
| 11.30% | 11.00% | 0.30% | 2.76% |
| 18.74% | 18.50% | 0.24% | 1.32% |
| 32.57% | 32.80% | 0.23% | 0.69% |

11 a. Beam diameter effects on haze measurements



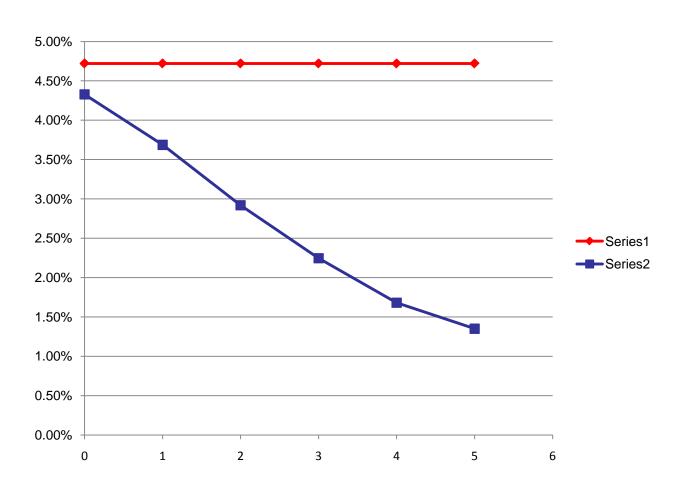
11 b. Beam diameter effects on haze measurements

| Entrance b | eam d=15 m | ım, exit b. D: | =22 mm | Entrance b | eam d=11 n | nm, exit b. D: | =17.5 mm |
|---|------------|----------------|-------------|--------------------------------------|------------|----------------|-------------|
| Haze(%) | H(stand) | Abs. error | Reltv error | Haze(%) | H(stand) | Abs. error | Reltv error |
| 1.68% | ú 1.60% | 0.08% | 5.23% | 1.58% | 1.60% | 0.02% | 1.09% |
| 4.63% | 4.72% | 0.09% | 1.90% | 4.36% | 4.72% | 0.36% | 7.66% |
| 10.40% | ú 11.00% | 0.60% | 5.48% | 10.35% | 11.00% | 0.65% | 5.93% |
| 17.85% | 6 18.50% | 0.65% | 3.52% | 17.24% | 18.50% | 1.26% | 6.79% |
| 30.67% | 32.80% | 2.13% | 6.50% | 30.22% | 32.80% | 2.58% | 7.86% |
| Average Er | rror= | 0.71% | 4.53% | Average E | rror= | 0.97% | 5.86% |
| Entrance beam d=8 mm, exit b. D=13.5 mm | | | | Entrance beam d=5 mm, exit b. D=9 mm | | | |
| Haze(%) | H(stand) | Abs. error | Reltv error | Haze(%) | H(stand) | Abs. error | Reltv error |
| 1.56% | ú 1.60% | 0.04% | 2.43% | 1.40% | ú 1.60% | 0.20% | 12.67% |
| 4.28% | 4.72% | 0.44% | 9.32% | 4.56% | 6 4.72% | 0.16% | 3.42% |
| 9.93% | ú 11.00% | 1.07% | 9.77% | 10.13% | ú 11.00% | 0.87% | 7.88% |
| 17.47% | 6 18.50% | 1.03% | 5.57% | 16.69% | 6 18.50% | 1.81% | 9.80% |
| 30.36% | 32.80% | 2.44% | 7.45% | 30.26% | 32.80% | 2.54% | 7.73% |
| | | 1.01% | 6.91% | | | 1.12% | 8.30% |

12. Future work

- 1. Study repeatability.
- 2. Study ways to measure thick samples:
 - study what happens when you make the beam smaller and larger;
 - position samples at various distances from the entrance port of the integrating sphere: 1", 2", 3", 4" & 5".
 - 3. Use empirical data to develop an equation showing how haze values change with the distance from the entrance port of the sphere.
 - 4. Find an approximate formula that would compensate for the change in haze values as the distance from the entrance port of the sphere increases from 0 to 5".

13. Dependence of the haze value on the sample distance from the port



14. Comparison of haze measurements with new values of standards obtained with the commercial tool and regression analysis

| SUMMARY OUTPUT | г | | | | | |
|----------------|--------------|----------------|-----------|-------------|----------------|-----------|
| Regression | n Statistics | | | | | |
| Multiple R | 0.9997771 | | | | | |
| R Square | 0.9995543 | | | | | |
| Adjusted R Sq. | 0.9994058 | | | | | |
| Standard Error | 0.0030402 | | | | | |
| Observations | 5 | | | | | |
| | | | | | | |
| ANOVA | | | | | | |
| | df | SS | MS | F | Significance F | |
| Regression | 1 | 0.062191023 | 0.062191 | 6728.438062 | 3.99362E-06 | |
| Residual | 3 | 2.7729E-05 | 9.243E-06 | | | |
| Total | 4 | 0.062218752 | | | | |
| | | | | | | |
| | Coefficients | Standard Error | t Stat | P-value | Lower 95% | Upper 95% |
| Intercept | -0.0038436 | 0.00219246 | -1.753108 | 0.177866968 | -0.010821004 | 0.003134 |
| X Variable 1 | 1.122452 | 0.013683924 | 82.027057 | 3.99362E-06 | 1.078903634 | 1.166 |

15. Comparison of haze measurements with new values of standards obtained with the commercial tool and regression analysis

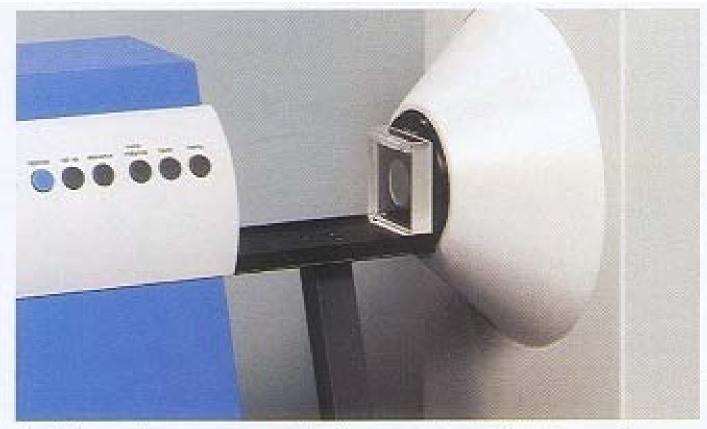
| Haze | Haze | Haze | Haze | m2 | b |
|-----------|----------|----------|--------------|------------------|----------|
| standards | expected | measured | adjusted | 1.122451986 | -0.00384 |
| X | Y0=X | Y=X1 | Y2=m2*X1+b E | Error=ABS(Y2-Y0) | |
| 1.60% | 1.60% | 1.53% | 1.33% | 0.27% | |
| 4.72% | 4.72% | 4.50% | 4.67% | 0.05% | |
| 11.00% | 11.00% | 10.41% | 11.30% | 0.30% | |
| 18.50% | 18.50% | 17.04% | 18.74% | 0.24% | |
| 32.80% | 32.80% | 29.36% | 32.57% | 0.23% | |
| | | A | verage Error | 0.22% | |
| | | W | orst Case | 0.30% | |

16. Back-up slide: Sample holder guided in a precision track system



Exact positioning of films with a holder guided in a precision track system.

17. Back-up slide: Cuvette table that could be used to hold heavy samples



Liquids are best measured using cuvettes and the cuvette table.